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AFRPL-TR-66-347

**RESEARCH ON INHIBITED  $N_2O_4$** **First Quarterly Report**

H. E. Dubb  
A. D. Lev  
J. Halchak

Rocketdyne  
A Division of North American Aviation, Inc.  
Canoga Park, California

**TECHNICAL REPORT AFRPL-TR-66-347**

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AFRPL-TB-66-347

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First Quarterly Report

H. E. Dubb  
A. D. Lev  
J. Halchak

December 1966

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## FOREWORD

The research reported herein was supported by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, Air Force Systems Command, United States Air Force, under Contract F04611-67-C-0008, with Ralph Fagnoli, 1st/Lt/USAF, RPCL, serving as Project Monitor.

The work described covers the period 1 September 1966 through 30 November 1966. The Responsible Scientist for this program is Dr. Hubert E. Dubb of the Analytical Chemistry Group, headed by Dr. B. L. Tuffly. The work was conducted by members of the Composition Research Unit, supervised by Dr. V. H. Dayan; the Propellant Engineering Group, headed by Dr. J. J. Kalvinskas; and the Materials and Processes Section, managed by Mr. G. A. Fairbairn.

This report has been assigned the Rocketdyne report No. R-6831-1.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. EHELKE, Colonel, USAF  
Chief, Propellant Division

# ABSTRACT

This program is concerned with extending the engineering evaluation of the new storable liquid oxidizer INTO which is NTO inhibited with 1 to 3 weight percent  $\text{PNO}_2$ .

Storability tests will be conducted on INTO stored at ambient temperature and at 70 C in aluminum, iron, and titanium tanks of 10- to 20-gallon volume. All necessary equipment for these tests is presently available or has been ordered.

Small-scale storability tests are to be performed on INTO stored at ambient temperature, at 70 C, and at 130 C in iron and titanium bombs of 20-milliliter volume. All of the equipment has been assembled for these tests.

Titanium 6Al-4V specimens stressed to 90,000 psi at ambient temperature and at 70 C will be observed for possible failures. The bar stock for fabricating the stress frames and stress specimens has been ordered. The design of the tanks in which the stressed specimens will be stored has been completed.

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## INTRODUCTION

The use of nitrogen tetroxide (NTO), the most widely used storable liquid oxidizer in the United States, has been continually hampered by corrosion problems. Dry NTO is not a highly corrosive liquid when in contact with most common metals of construction, but moist NTO is extremely corrosive because of the formation of nitric and nitrous acids by the reaction of NTO with water.

It has previously been demonstrated under Contract AF04(611)-10809 (Ref. 1 through 3) that the addition of a fluorine oxidizer to NTO leads to a reduction of the nitric and nitrous acid content of the propellant with the concurrent production of HF. It has also been shown (Ref. 2 and 3) that if the fluorine oxidizer is  $\text{FNO}_2$ , the resulting oxidizer system is storable at 70 C in passivated aluminum, stainless-steel, and nickel containers.

The present program consists of an extended engineering evaluation of INTO, which is NTO containing 1 to 3 weight percent  $\text{FNO}_2$ . INTO is being evaluated with respect to its storability at ambient temperature and at 70 C in large tanks of 10- to 30-gallon capacity (Task I), and at ambient temperature, 70 C, and 130 C in small bombs of 20-milliliter capacity (Task III). It is also being evaluated at ambient temperature and at 70 C with respect to its effect upon the stress corrosion problem encountered when NTO is stored under pressure in titanium 6Al-4V tanks (Task II). The results of the program to date are presented in the following sections.

## TASK I: LARGE-TANK STORAGE

### SUMMARY

Under the previous contract, AF04(611)-10809, storability tests were conducted in 10-milliliter containers of stainless steel, nickel, and aluminum. The promising results of these tests (Ref. 1) indicated that further study should be made in this area. The objectives of this task are to investigate the storability of inhibited NTO (INTO) in approximately 10-gallon tanks and to investigate the formation of  $\text{FNO}_2$  (the inhibiting agent) in the tanks in situ. The results of these tests should be applicable to field conditions.

### EXPERIMENTAL

The storability of wet INTO (1 to 3 weight percent  $\text{FNO}_2$  formed in NTO containing 0.1 to 0.3 weight percent  $\text{H}_2\text{O}$  equivalent) will be conducted in approximately 10-gallon tanks of the following alloys:

1. Type 347 stainless steel
2. Type 1018 carbon steel
3. 18 percent nickel-maraging steel
4. Type 2219 aluminum
5. Type 2014 aluminum
6. Titanium 6Al-4V

One tank of each alloy will be stored at both ambient temperature and at 70 C for 12 months. A sampling valve will be included on each tank enabling liquid samples to be taken. Infrared analysis will be performed on samples to ascertain the original formation of  $\text{FNO}_2$  and to detect any  $\text{FNO}_2$  concentration change with time. Liquid samples may also be analyzed on an atomic absorption spectrophotometer to determine the dissolved metallic ion concentrations both at the beginning and end of the storage period.

### Facility Buildup

This task will be performed in Building B of the Polymer Development Area at Rocketdyne's Santa Susana Field Laboratory. A floor plan of the building with anticipated facility layout is presented in Fig. 1. The building is of corrugated sheet metal construction and is divided into two bays by an 8-foot metal partition. A loading facility will be installed in one bay and a storage and sampling facility will be installed in the other. Garage doors provide free access to each side. An overhead firex system will automatically provide a water deluge to both bays in case of a major release of INT0 from any of the storage tanks.

A loading facility has been designed and is currently under construction. A schematic of this facility is presented in Fig. 2.

### Experimental Procedures

A typical loading operation (with reference to Fig. 2 ) will be carried out as follows:

1. The tank, fluorine fill lines, and NTO fill lines will be evacuated.
2. Wet NTO will be loaded into the tank by  $\text{GN}_2$  pressurization of the NTO reservoir (the desired weight of NTO will be registered on the scale).
3. The fluorine loading volume will be brought to a pressure of approximately 50 psi from the main fluorine reservoir.
4. Fluorine will be bubbled slowly through the NTO (via the dipleg) until the required concentration of  $\text{FNO}_2$  (determined by infrared analysis) is reached.

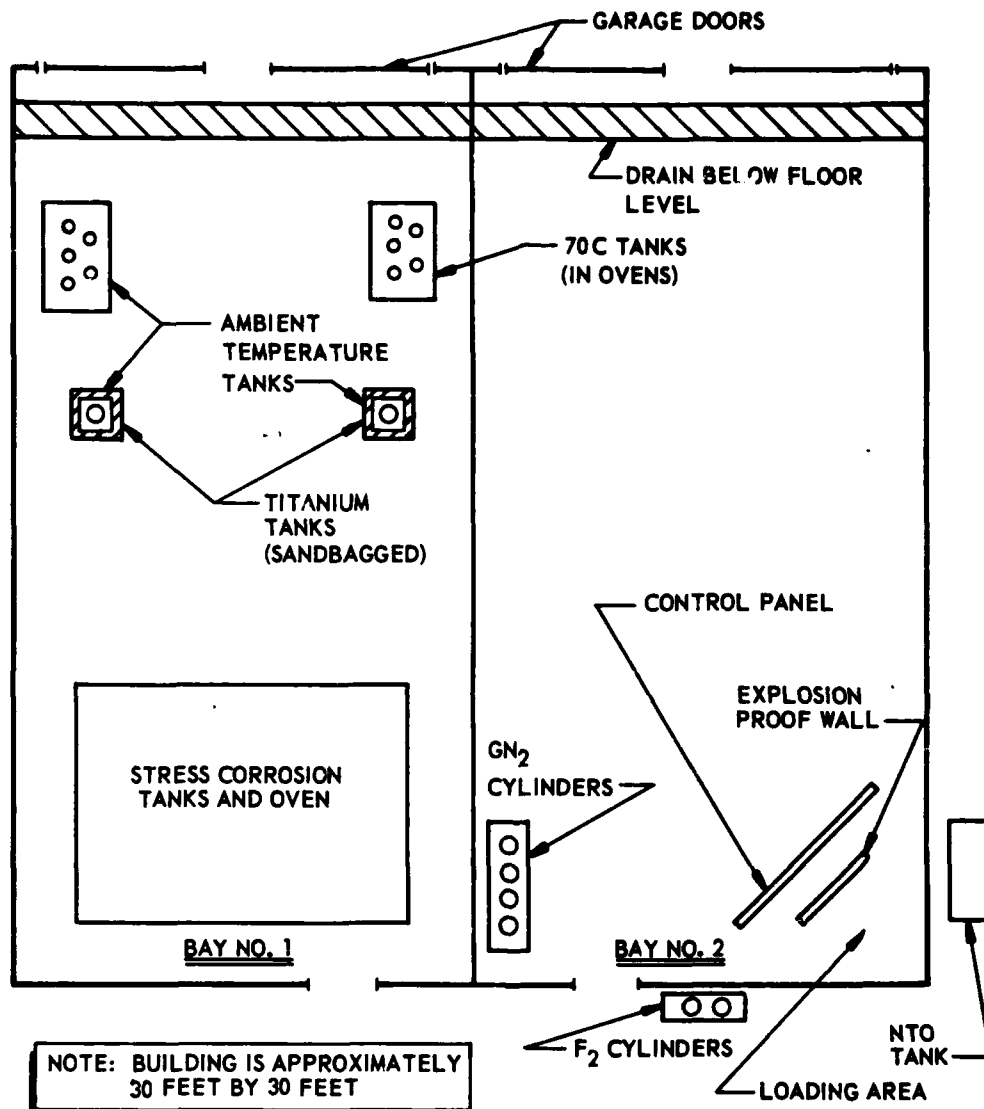


Figure 1. Floor Plan of Building B

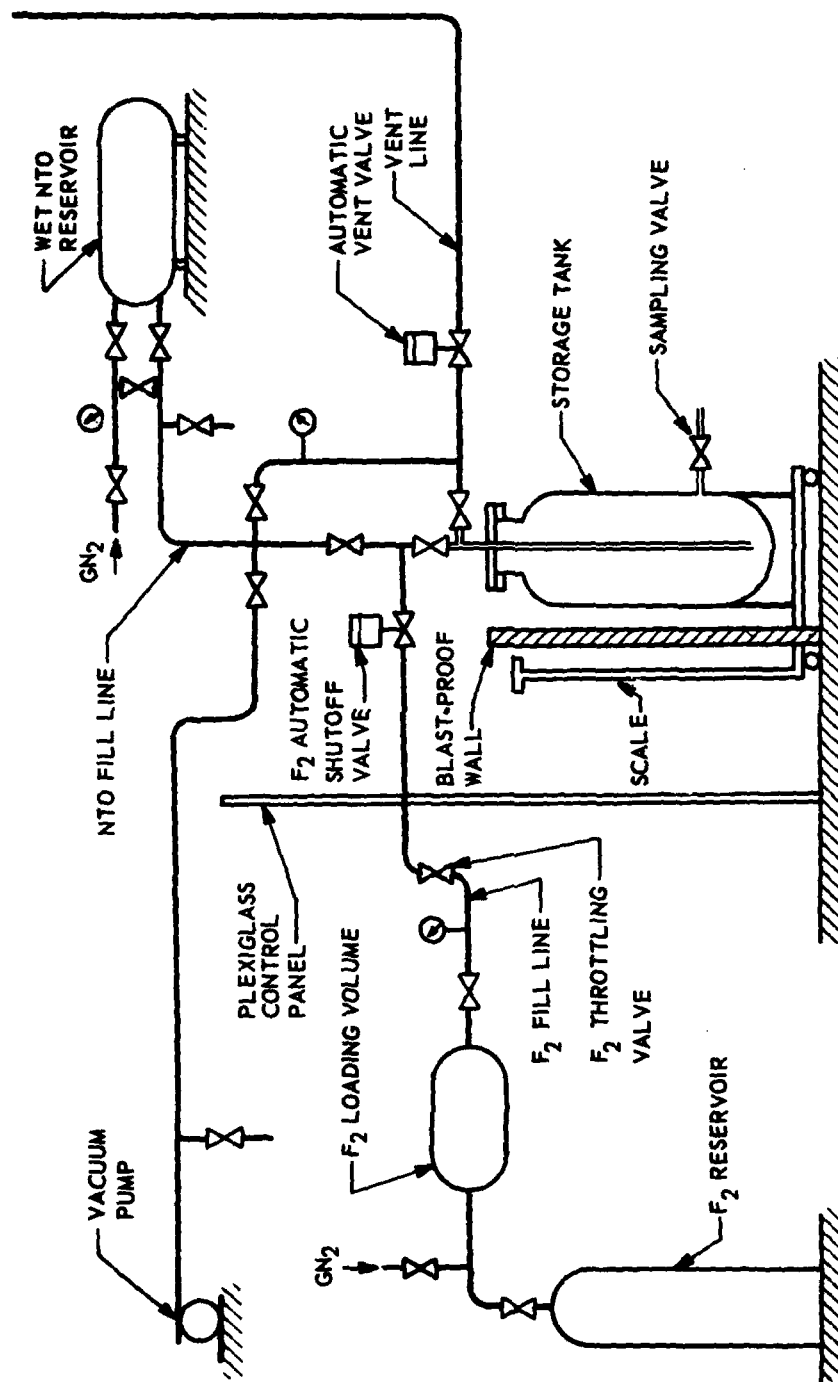


Figure 2. Loading Facility Schematic

Infrared samples will be taken on a specially designed portable sampling rack. This rack, attached to the tank sampling valve, is illustrated in Fig. 3. The sampling operation will be effected as follows:

1. The sampling system (which has been prepassivated with  $\text{FNO}_2$ ) will be evacuated.
2. A liquid sample will be trapped between the tank sampling valve and the expansion valve.
3. The sample will be expanded into the infrared cell until the desired pressure is reached.
4. Excess liquid sample will be transferred to the sample dump cylinder.
5. The gas sample will be analyzed on an infrared spectrometer.

All tanks will be stored in Bay No. 2 of Building B (Fig. 1). The garage door will be open to expose the six ambient temperature tanks to actual Santa Susana ambient temperatures. The high-temperature tanks will be stored in ovens constructed of sheet metal and duct insulation. The ovens will be set on firebrick bases to minimize heat losses. Temperature will be held constant by thermostatically controlled heating elements. For both ambient and high-temperature tests, the titanium tanks will be isolated from the other five alloys and surrounded with sandbags. This will be done because of the possibility of stress corrosion failure in the case of titanium.

#### Storage Tank Procurement

Carbon steel, maraging steel, and aluminum 10-gallon tanks have been designed by the Facilities Engineering Group at Rocketdyne and are currently under construction by a local fabricator. The tanks are illustrated in Fig. 4 as they will appear when ready for loading. The tank fittings

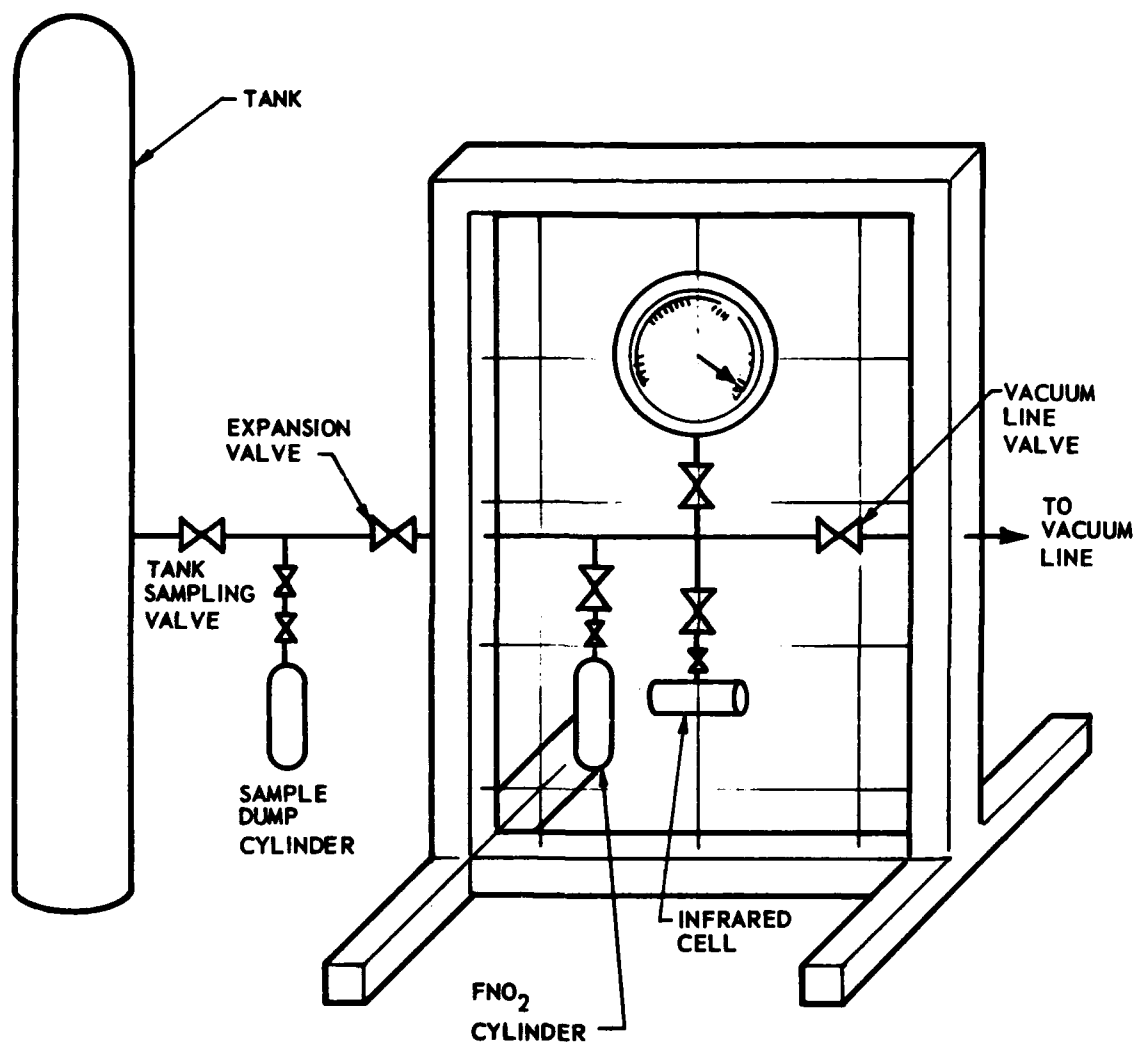


Figure 3. Portable Sampling Rack Schematic

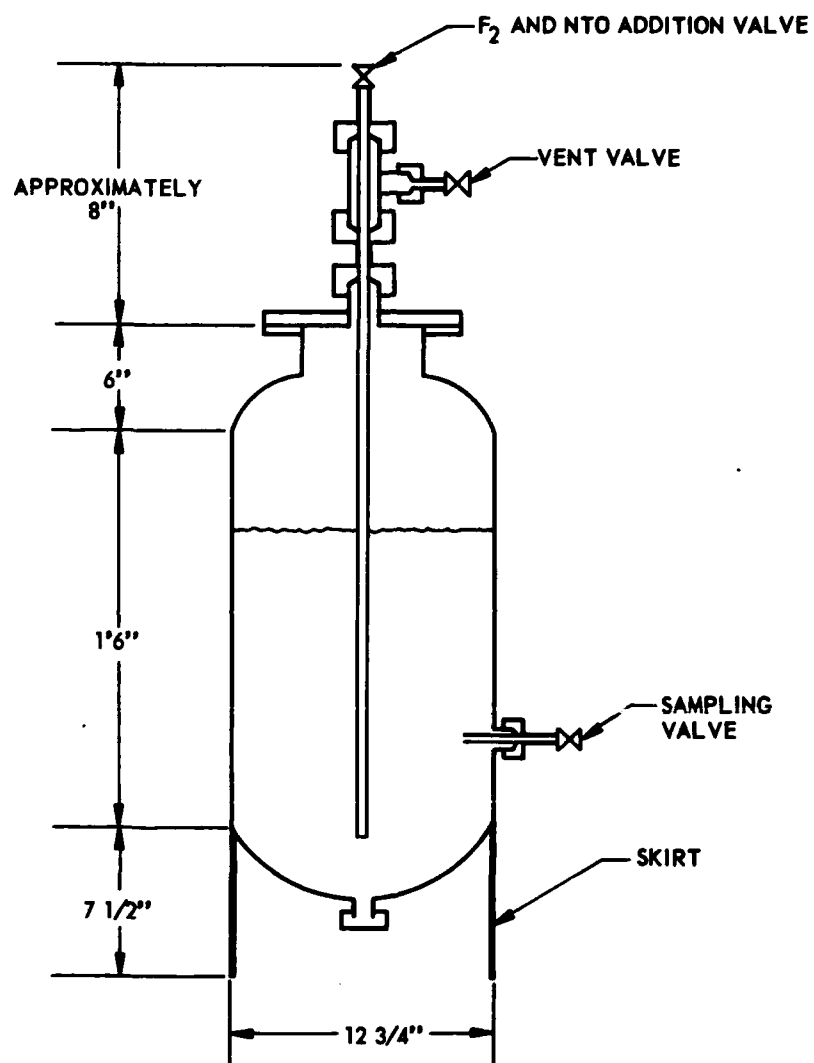


Figure 4. Storage Tank Schematic



have been designed to facilitate loading of NTO and fluorine through one port at the top, and tank ullage venting. The sampling port fitting extends 3 inches into the tank to ensure that all samples taken will be from the bulk volume of INT0. The tanks will be flanged to facilitate cleaning and inspection both before and after the program. Tanks are scheduled to be delivered to Rocketdyne by 9 February 1967.

The Type 347 stainless-steel tanks are ready to be loaded, and the titanium tanks are scheduled to arrive at Santa Susana in the near future. The stainless-steel tanks have an 18-gallon capacity and are of the same general configuration as the 10-gallon tanks. The titanium tanks are 22-inch spheres with an approximate 30-gallon capacity. They will be fixed with a flange on which loading and sampling fittings similar to those of the 10-gallon tanks will be installed.

Loading of the stainless-steel tanks will commence as soon as the facility is completed. The titanium, carbon steel, aluminum, and maraging steel tanks will then be loaded in that order. The loading and sampling of the tanks is not expected to pose any major technical difficulties.

## TASK II: TITANIUM STRESS CORROSION

### SUMMARY

Designs have been completed and Ti-6Al-4V bar stock has been ordered for stress-corrosion tensile bars and stressing frames; the material is scheduled for delivery by 15 December. Work performed during a previous investigation revealed no problems with all-titanium stressing frames. Initial design work on the Ti-6Al-4V pressurization tank is complete and a fabrication source is being sought.

### EXPERIMENTAL

For the stress-corrosion phase of the program (Task II), Rocketdyne-designed stressing frames and 0.125-inch-diameter tensile specimens will be employed, as illustrated in Fig. 5 through 7. Uniaxial tensile stress may be applied to the specimens by torquing the two bolts at the top of the stressing frames. Similar stressing techniques have been used extensively, at Rocketdyne, for the study of stress corrosion cracking in aluminum alloys. Experience has shown that these techniques will yield both reliable and reproducible stress corrosion data. The amount of strain imparted to the tensile specimen is measured with a 1/2-inch extensometer; knowing the strain, the stress on the specimen can be calculated using the tensile elastic modulus of the material ( $\sigma = \epsilon E$ ), provided the proportional limit is not exceeded. In practice, specimens are strained a predetermined amount, which has been calculated to produce the desired level of stress. To eliminate errors caused by variances in the calibration of extensometers and recording equipment, published values of elastic modulus are not employed. Instead, several tensile specimens, from the same lot of material to be used during the stress-corrosion tests, are tensile tested as control specimens. The value for the elastic modulus is obtained from the stress-strain curves of the control specimens; the same extensometer is then used in loading the remaining specimens in the stressing frames.

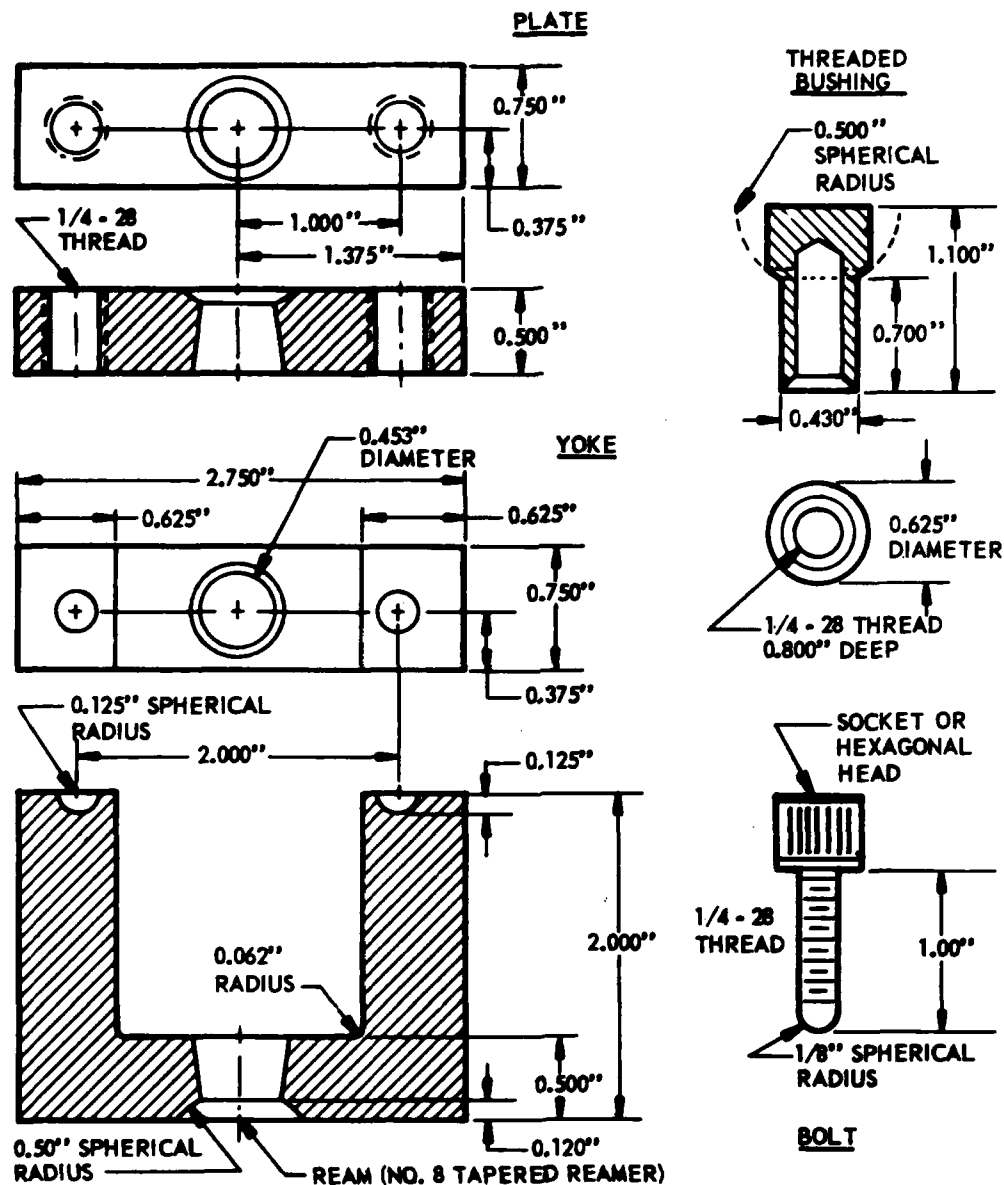


Figure 5. Stressing Frame Schematic

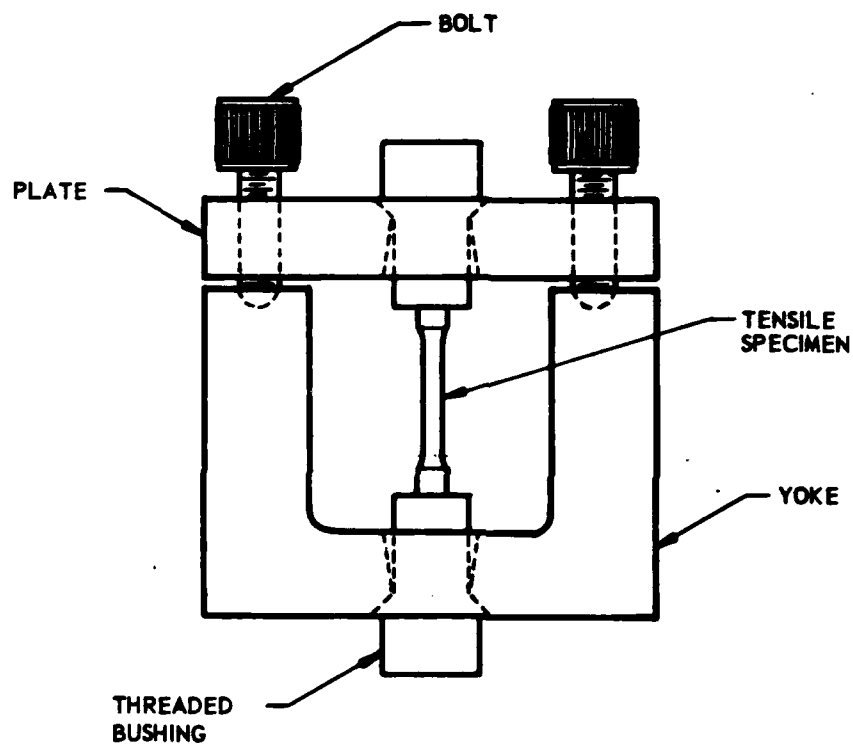


Figure 6. Schematic of Assembled Stressing Frame

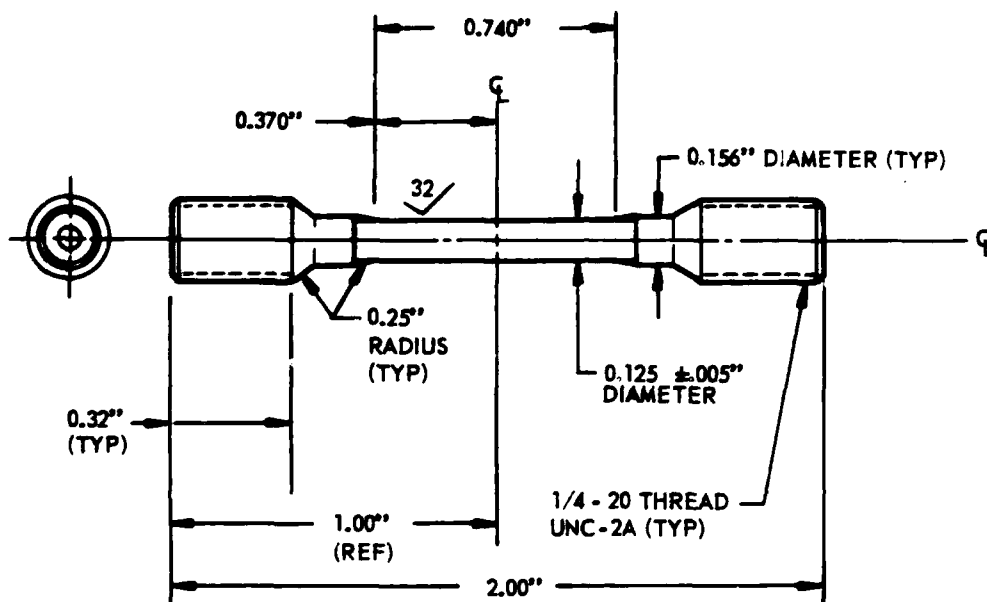


Figure 7. Stress Corrosion Tensile Specimen

During this program, all material used in the stressing frames will be the same as the tensile specimens (Ti-6Al-4V in the solution treated and aged condition). Five all-titanium stressing frames were used during a previous stress-corrosion investigation and no problems with seizing or galling of the titanium bolts were encountered, although no lubricant was employed on the threads.

Some difficulty was experienced in ordering the raw material because of the general shortage of titanium alloy raw stock. The originally desired sizes of bar stock were not available; therefore, compromise sizes were ordered. To date, 1- and 3-inch-square bar stock has been ordered for tensile specimens and stressing frames, respectively. The material is standard grade Ti-6Al-4V in the annealed condition, conforming to Rocketdyne material specification RA0170-054. Delivery is scheduled for 15 December. After receipt of material, rough cuts will be made to produce sizes suitable for heat treatment. The stock will be solution heat treated and aged per specification RB0111-024 (1750 F, 1 hour water quenched and 1000 F, 8 hours air cooled) to a minimum ultimate tensile strength of 160,000 psi and yield strength of 150,000 psi.

A schematic of the pressurization tank, which will contain the propellants and stress-corrosion specimens, is presented in Fig. 8. Two tanks will be required, each will be built entirely of Ti-6Al-4V, and will be required to contain 200-psig pressure at a safety factor of 5 to 1.

Present tank design consists of a 9-inch-diameter by 10-inch-long cylinder with hemispherical end caps. A bolted flange will be provided at one end for loading and unloading specimens. Minimum wall thickness will be 0.040 inch, which easily furnishes a 5 to 1 safety factor.

Tank walls will be stressed to 22,500 psi at a maximum operating pressure of 200 psig. The tank will be fabricated by TIG welding Ti-6Al-4V in the solution-treated condition. Following welding, the tanks will be aged 6 to 8 hours at 1000 F; aging will serve the dual purposes of strengthening

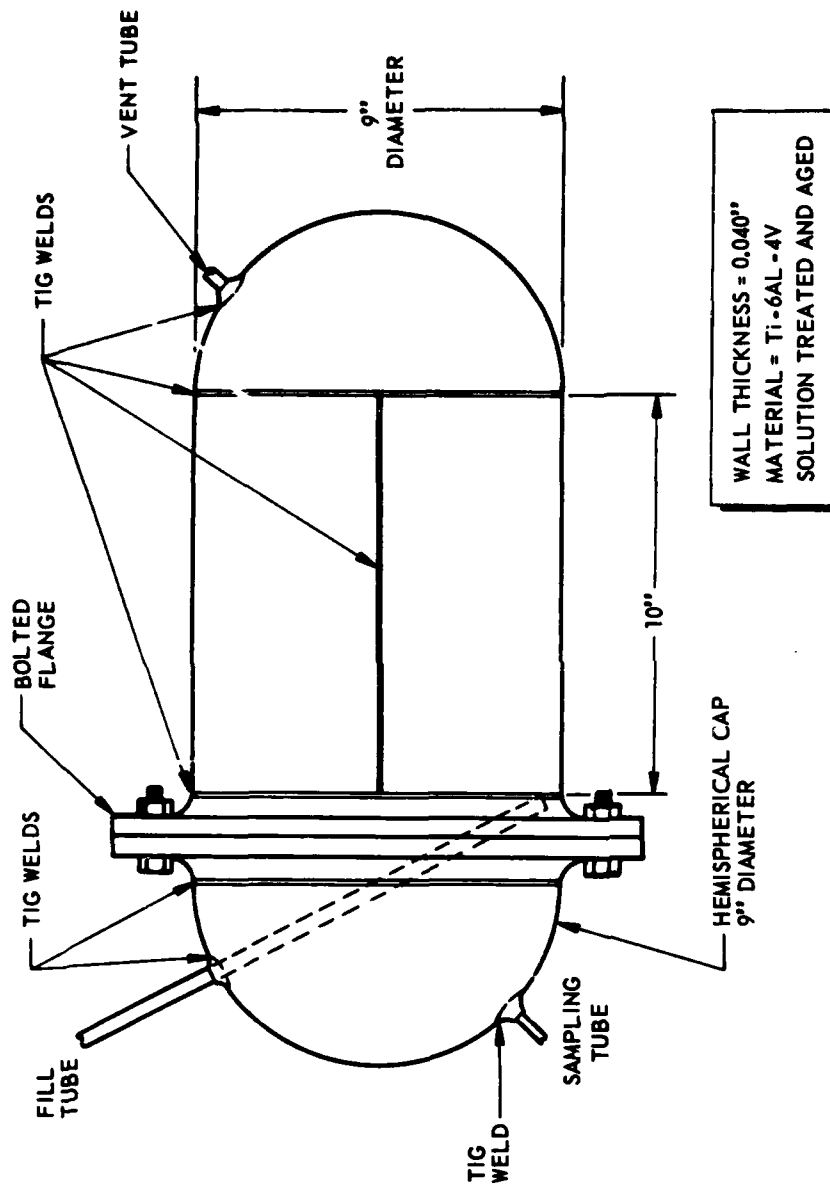


Figure 8. Pressurization Tank Schematic

the parent metal and of stress relieving the weldments. During aging, the tanks will be purged on the inside with argon to prevent formation of the blue oxide interference film on the surface of the inner walls. Pressure vessel fabricators are now being contacted for bids; to reduce costs, maximum aging will be made of already existing shapes and sizes, this may necessitate minor modification of the present tank design.

#### CONCLUSIONS

The specimens will be stored at ambient temperature and at 70 C for periods of 60 days. The specimens will be examined by radiography every 3 to 4 days for the first 2 weeks and every 5 days thereafter. The rate of failure of the samples will be compared to the rate of failure of similar specimens exposed to NT0 where such data is available. If failures occur, metalographic, fractographic, electron probe, and chemical analysis of the fractured specimens will be performed in an attempt to determine the mechanism of the failures.



### TASK III: SMALL-BOMB STORABILITY

#### SUMMARY

Small-bomb storability tests will be conducted with INT0 prepared from dry NT0 (< 0.10 weight percent  $H_2O$ ) and of INT0 prepared from wet NT0 (> 0.10 weight percent  $H_2O$ ). The tests will be conducted at ambient temperature and at 70 C for 12 months and at 130 C for 24 hours. The bombs are fabricated of three materials: (1) titanium 6Al-4V, (2) Type 250 maraging steel, and (3) Type A286 steel welded with Hastelloy-W rod. They have an internal volume of 20 milliliters. The bombs have been assembled, cleaned, and tared. A vacuum system has been modified to allow the introduction of approximately 18.5 milliliters of INT0 into each bomb. A supply of 300 grams of INT0 has been prepared to passivate the bombs.

#### EXPERIMENTAL

The system which will be used to load the bombs is illustrated in Fig. 9. The bomb will be attached to the system at AN fitting No. 3 with all valves shut. Valve No. 2 will be opened to the vacuum line and valves No. 3 through 5 will also be opened. The system will then be pumped until a vacuum is attained between valve No. 1 and the end cap. The vacuum pump will be isolated from the vacuum system, the loading assembly will be allowed to stand for 30 minutes, and the Heise gage will be observed to determine if there is any leakage. If there is a pressure increase, the leak or leaks causing the increase will be detected and repaired. Loading will proceed when the system is leak free.

Valves No. 2, 3, and 5 will be shut off. Valve No. 1 will then be opened. Time will be allowed for propellant from the 1-liter Hoke reservoir to fill the 18.5 milliliter volume between valves No. 1 and 3. Valve No. 1 will then be closed and valve No. 3 will be opened. Time will be allowed

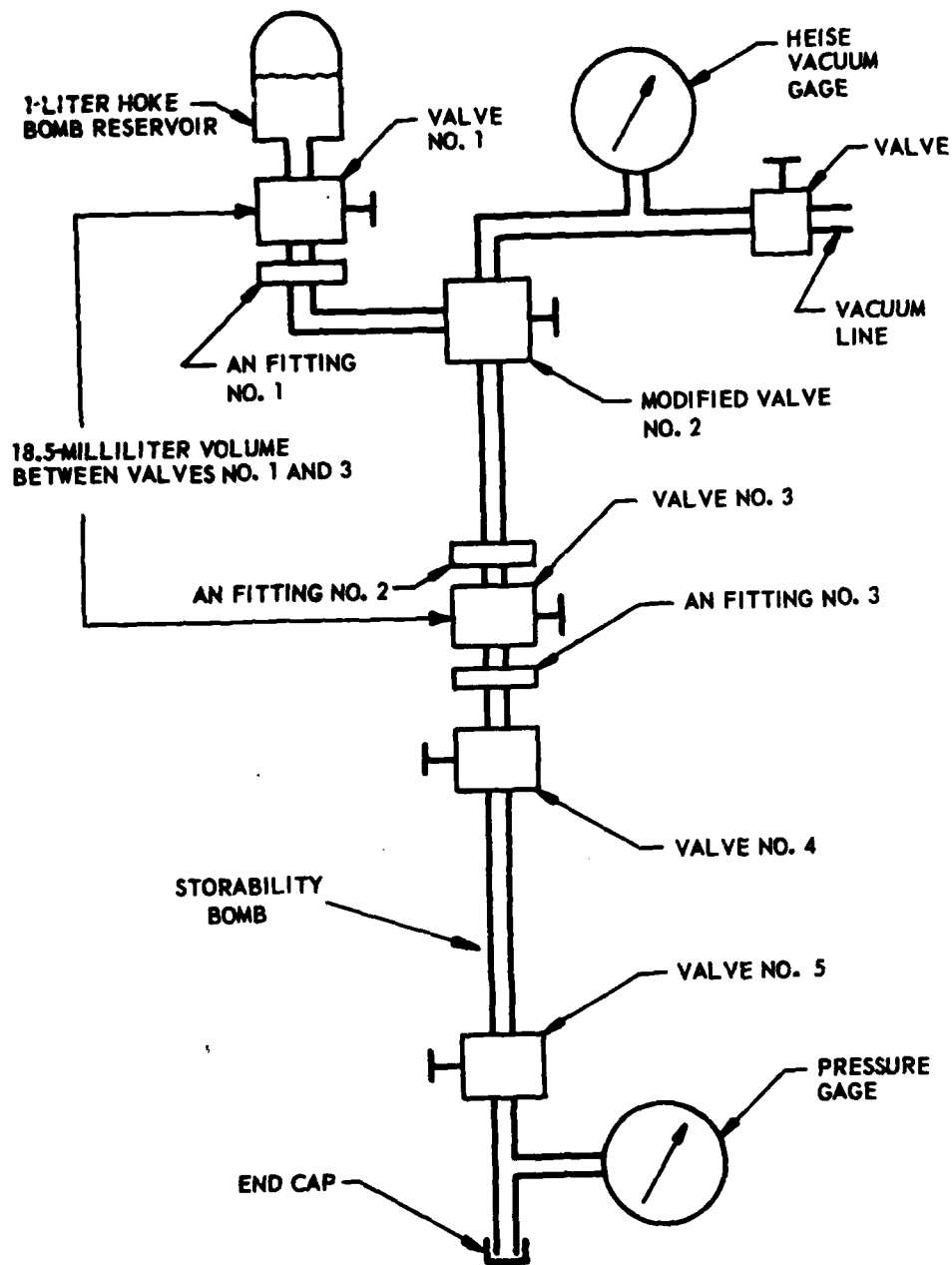


Figure 9. Small-Bomb Loading Assembly

for the liquid trapped between valves No. 1 and 3 to flow into the storability bomb. Valve No. 4 will then be closed. Any excess vapor or liquid still contained between valves No. 1 and 4 will be pumped into a -196 C trap on the vacuum line for later disposal. Valve No. 3 will then be closed. The storability bomb will be disconnected at AN fitting No. 3 and inverted. Valve No. 5 will then be opened and the pressure will be read. Then valve No. 5 will be closed and the bomb will be introduced to either an ambient, 70 C, or 130 C temperature environment. The pressure in the bombs will be checked periodically by adjusting the temperature to 25 C, and opening valve No. 5. Sampling to determine the composition of the liquid will be accomplished through valve No. 4.

#### CONCLUSIONS

The conclusions which will be reached as a result of the experimental program outlined previously will be an evaluation of the storability of INTO in the three materials being tested. The storability will be evaluated with respect to possible changes in  $\text{FNO}_2$  content and in pressure.

## SUMMARY

All equipment for the Task I large-tank storage tests is presently available or has been ordered. The vacuum line for loading the tanks is under construction. Safety features of the building in which the tests are to be conducted have been modified to suit the requirements of the program.

The necessary materials for the Task II titanium stress corrosion study have been ordered. The tanks in which the stressed specimens will be stored are being designed.

All of the equipment for the Task III small-scale storage tests has been received. The bombs have been assembled and weighed. An INTO solution has been prepared for passivating the bombs.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nitrogen Tetroxide						
Fluorine oxidizer						
Storability						
Stress Frames						
Stress Specimens						
Aluminum						
Iron						
Titanium						
Vanadium						

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There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

**14. KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.

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